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The study of atomic and molecular adsorption on well defined metal surfaces is of crucial importance to the understanding of many technologically important processes such as thin film growth, catalysis, corrosion, and the rates of chemical reactions on surfaces. The experimental methods employed in this study include the field ion microscope (FIM) and hyperfine interaction techniques. The systems chosen for study included the adsorption of hafnium and cadmium atoms on single crystal surfaces of tungsten and nickel. The choice of experimental techniques and systems for this study was dictated by a desire to evaluate hyperfine interaction techniques for the

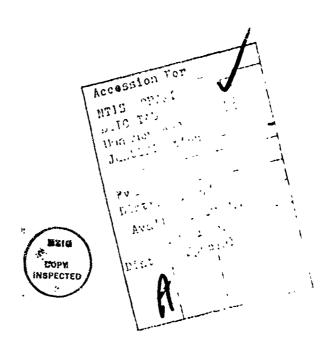DD FORM 1473 EDITION OF 1 NOV 65 IS OBSOLETE

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FINAL REPORT

Objectives: The study of atomic and molecular adsorption on well
defined metal surfaces is of crucial importance to the understanding
of many technologically important processes such as thin film
growth, catalysis, corrosion, and the rates of chemical reactions
on surfaces. The experimental methods employed in this study
include the field ion microscope (FIM) and hyperfine interaction
techniques. The systems chosen for study included the adsorption
of hafnium and cadmium atoms on single crystal surfaces of tungsten
and nickel. The choice of experimental techniques and systems
for this study was dictated by a desire to evaluate hyperfine
interaction techniques for the study of surface adsorption,
through comparison with the results obtained by the now well
established FIM technique for the study of surface adsorption.

Summary of Results: a) Field Ion Microscopy - Graham and Bajoria

Field ion microscope studies of adsorption and diffusion of
hafnium and cadmium on tungsten (111), (211), and (321) surfaces
have been completed. Hafnium atoms are adsorbed only in normal
lattice sites on W(111), while in contrast cadmium atoms are
observed in both normal lattice sites and bridge sites. The
diffusion characteristics, including activation energies for
diffusion and observation of the crystallographic dependence of
diffusion dimensionally have been studied for Hf and Cd single
adatoms on tungsten (111), (211), and (321) and are summarized in
Table 1. The effects of hydrogen on the onset temperature for

surface diffusion has also been examined. Details of these studies are in preparation for publication. These publications are entitled:

"Adsorption and Diffusion of Hafnium on Tungsten Surfaces", and "Adsorption and Diffusion of Cadmium on Tungsten Surfaces", by D. J. Rajoria and W. R. Graham.

TABLE 1

System Absorbate/Substrate	Diffusion Onset Temp.(K)	Activation Energy(eV)	Dimensionality of Diffusion
Hf/W(211)	221	.54	1D
Hf/W(321)	186	.57	1D
Hf/W(111)	>600	>2	
Cd/W(111)	257(normal) 136 bridge	(to be calculated) " " "	2D
Cd/W(211)	92	" " "	2D

b) Perturbed Gamma-gamma Angular Correlation - Caspari and Weiner

The purpose of the angular correlation portion of the experimental program was to determine the magnitude, direction and assymetry of the electric field gradient at the nucleus of a Cd^{111m} probe atom which is placed on the surface of a single crystal of tungsten or copper. The object was to determine from this information the geometric characteristics of the surface site or sites occupied by the probe atoms as a function of temperature, and crystallographic orientation of the surface plane.

This type of geometric information is at present not attainable by any other experimental technique.

This is the first time to the best of our knowledge, that an angular correlation has been attempted on any clean crystallographic surface. In order to conduct the experiment an angular correlation facility capable of surface studies was constructed from scratch. The principal requirements of such a system are:

1) Radioactive probe atoms with relatively short halflives must be used, since in a surface study one must collect sufficient counts to determine the geometric configuration in the relatively short time available while the surface stays clean (typically 1-3 hours) and do this with a fraction of a monolayer coverage so that the majority of probe atoms are isolated adatoms with the nearest and next nearest neighbor surface sites vacant. In our case the halflife of Cd^{111m} (our probe atom) is about 48 minutes, so that in order for it to be available on site it has to be produced on site. We solve this problem by producing the probe atoms using the Tandem accelerator situated on the University of Pennsylvania campus, by bombarding a palladium foil with 24 MeV alpha particles and inducing the $Pd^{108}(\alpha,n)$ reaction.

2) The probe atoms in the palladium matrix must be inserted into an ultrahigh vacuum chamber where the probe atoms can be deposited on the tungsten or copper substrate and where the angular correlation experiment can be carried out. Due to the short halflife of the probe atoms, this process should take no longer than a few minutes. During the insertion the substrate surface must either remain clean or be quickly cleanable. This imposes the requirement

that when the probe atoms are injected into the main vacuum chamber they must not contaminate the substrate surface. In our case this problem is handled by first introducing the probe atoms, which are still embedded in their palladium matrix where they were created, into a small vacuum chamber which is constructed adjacent to the main chamber and connected to it through a valve.

This small chamber is temporarily brought up to atmospheric pressure while the probe atoms embedded in their matrix are transferred from the vacuum system of the Tandem accelerator to a track assembly inside the small vacuum chamber, which is then quickly sealed and pumped down to about 10^{-9} torr. During and after the pumpdown procedure the palladium is cleaned by passing a current through the foil using contacts made through a special bay which is in contact with the track assembly at this stage. After cleaning and pumpdown have been completed, the valve between the chambers is opened and the probe atoms in their matrix are carried on the track assembly to another special bay in the main chamber using an injection rod. The rod is then withdrawn back into the small chamber which is subsequently sealed off and a vacuum of about 10^{-11} torr is regained in the main chamber.

3) Once the foil is in the main chamber with the substrate there must be an efficient method of depositing cleanly the probe atoms onto a clean target surface. The method we use for performing th cleaning of the substrate surface, deposition of probe atoms onto the substrate surface, angular correlation measurements (and any other procedure we may want to use in the future e.g. Auger measurements) is to move the crystal along a 60 inch track to five

stations in the vacuum chamber, where the various procedures and measurements can be carried out. The substrate surface can either be cleaned by ion bombardment at one of these stations, or it can be cleaned by passing a current through special connectors on the crystal mount. This mount is not only designed to permit electrical conduction heating of the substrate surface, but can also be connected to a liquid nitrogen reservoir with a connection of sufficient thermal conductivity to allow us to cool the substrate surface to a temperature low enough so that when the probe atoms are deposited onto the surface their sticking coefficient will be high enough so that they will not boil off from the surface during the experiment. When the substrate surface is clean and sufficiently cool it is then placed very near to the probe atoms in their matrix and the probe atoms are then deposited onto the substrate surface by conduction heating the palladium matrix to about 900°C. At this temperature the matrix itself is stable with a very low vapor pressure, however the Cd^{111m} probe atoms migrate to the surface of the foil and are thermally ejected. We have successfully removed 75% of the probe atoms in this fashion.

4) Once the experimental system has been created it is then necessary to perform the angular correlation. In order to take full advantage of the potential information available from gamma-gamma perturbed angular correlations a very powerful but simple apparatus has been put together. Since we are working with single crystal surfaces we can regard the principal axis system of the probe atom as lying in a unique direction with respect to the directions of the first and second gamma rays used in the

correlation. Therefore the correlation has intrinsicly four angular degrees of freedom which are independently specifiable. In order to accommodate this requirement a goniometer was built which allows each of two system detectors to be independently placed anywhere over a full quadrant of the sphere, ranging from a substrate surface orientation of 0° to 90° and along any chosen azimuthal angle through a range of 180°.

Two constant fraction differential discriminators are then employed so that both timing and energy selection may take place in the same channel, thereby raising the upper limit on the achievable coincidence count rate by eliminating the need for a separate energy analysis channel and the associated slow coincidence unit that would be required in order to interface it with the timing channel.

A special high speed multichannel analyzer was designed to analyze and store the output from the time to amplitude converter which carries the information that a coincidence has occurred and the amount of time that the probe atom spent in the intermediate state. A surface angular correlation data collection system has two basic requirements that can not be met by 'store bought' multi-channel analyzers which are usually based on some microcomputer system. The first is that in order to accumulate sufficient statistics in a short amount of time the analyzer has to be capable of processing the incoming pulses at very high count rates, and secondly if during a given session one wants to take more than one set of data, then the analyzer has to be capable of emptying it's memory containing the previous data set within a few minutes. In order to meet both these basic requirements and also completely

integrate the information processing system a special high speed computer based 4096 multichannel analyzer was built for the facility. This analyzer is capable of analyzing and storing input pulses at over 200,000 cts/sec, which it achieves principally by using a hardwire TTL based computer which is controlled by a microprocessor in order to increment the appropriate element of memory, indicating a pulse has occurred with a certain height. The conventional technique in store bought systems is to use the microprocessor directly to increment memory. This technique, however limits the possible count rate to about 15-20 KHz. In addition, the analyzer is able to dump it's data at 9600 baud, which means that it only takes about four minutes to purge the system. Finally, the computer based multichannel analyzer also acts as a terminal and is linked to an IBM 4341 where the main data processing is done. This allows the data to be sent directly to the IBM 4341 during the experiment thus integrating the data processing function.

5) The theoretical expressions necessary in order to interpret the data produced during the experiment were derived. As was mentioned before, the correlation has four angular degrees of freedom, and although the principal axes system is thought to lie in a unique direction (e.g. principal axes perpendicular to the surface), the direction itself has to be determined from experiment. The theory also has to take into account the possibility that the electric field gradient may be assymetric. This sort of calculation is being done numerically for each point in time.

The experimental procedures and apparatus described above were essentially completed during the period covered by the contract.

Further work on the actual experiment using Cd probes on tungsten and copper substrates are still in progress. The experimental facility which has been developed represents a unique tool for the measurement of surface hyperfine interactions. Probes other than $Cd^{111}m$, as long as they have sufficient specific activity and suitable intermediate state lifetimes can also be used. In addition, while we have measured the hyperfine interaction using the technique of perturbed angular correlations, Mössbauer studies can also be made on our facility with only very minor modifications.

Adsorption of Hafnium on Tungsten Surfaces

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ABSTRACT

Field ion microscope studies of adsorption of hafnium on tungsten (111), (211) and (321) surfaces are reported. Hafnium adatom on W(111) are observed only in normal lattice sites. The presence of hydrogen lowers the onset temperature for diffusion of Hf on W(111). Single Hf atom on W(211) and (321) surfaces exhibit one dimensional channeled diffusion with activation energies .54±.06 eV and .57±.06 eV respectively. The activation energies for Hf diffusion on these planes follow a trend consistent with that reported for binding energies of the 5d transition metal series.

Introduction

The study of atomic and molecular adsorption on well defined metal surfaces is of crucial importance to the understanding of many technological processes such as thin film growth, catalysis, corrosion, and rates of chemical reactions on surfaces. In the case of metallic adsorption studies field ion microscopy demonstrates an added advantage in that the technique not only provides a direct means to study adatom site symmetry and the surface diffusion parameters but also allows the preparation of a single crystal planes of near perfect atomic order through field evapora-

tion at high fields and low temperatures[1]. In case of W and Ni

adsorption on the W(111) plane, the spatial resolution and magni-

fication of FIM makes it possible to see the adatom and underlying.

Substrate atom simultaneoulsy. In this paper we report the

results of hafnium adsorption on tungsten surfaces. The purpose

of this study was to investigate site geometry and diffusion para-

meters of metallic atoms, and especially those which have a radio-

active isotope such as Hf and Cd, and then to utilize the knowledge

obtained in this study to evaluate quantitatively the hyperfine

interactions investigated through perturbed angular correlation

measurements under ultra high vacuum.

Experimental

The FIM employed for this study has been described earlier[2].

After two 12 hr backouts at 300°C and standard degassing procedures

a system base pressure ~ 8×10^{-11} torr is achieved. The 40 mm

varian LSE microchannel plate used for the image intensification

is outgassed by electron bombardment for 12 to 24 hrs after each

backout to the point where no discernible pressure rise can be

detected by the ion gauge in the 10^{-11} torr range.

The specimen wires were .005" high purity single crystal

tungsten wires oriented along either the <111> or <211> directions[3].

A section of the specimen wire, about 0.15" long was spot welded

to a .007" diameter tungsten which was in turn mounted on two of

the four feed through pins of the cold finger. Two .003" diameter

tungsten potential leads are spot welded to the central section

of the specimen support loop. This four lead arrangement, in

conjuction with a temperature control circuit based on a kelvin
bridge, allows accurate control and measurement of the specimen
temperature.

A .010" diameter Hf wire was electropolished in 1.8N NaOH
solution at 5V a.c. and spot welded to a pair of tungsten support
loops. The support loops and the hafnium evaporation were
thorougly outgassed after each bake and the Hf filament was kept
at red heat between experiments to minimize the possibility of
contamination. This hafnium filament when resistively heated
served as an evaporation source of hafnium adatoms.

The image gas handling system includes provision for the use
of helium, hydrogen, neon or a controlled mixture of these gases.
High purity imaging gas from a glass lecture bottle is introduced
into the system at a constant flow rate. The gas inlet line also
contains a set of liquid nitrogen cooled tantalum getters to
further ensure the cleanliness of the gas. The tantalum filaments
were degassed thoroughly after each bake out.

Adatom Site Geometry and Surface Diffusion Procedures.

The .005" diameter tungsten specimen wire was electropolished
in 2N NaOH using a d.c. etch at approximately 10 V, producing
end forms with radii < 100A. The specimen was mounted in the
FIM and after UHV was achieved, initial field evaporation was
performed to obtain an end form of near perfect crystal structure.
The specimen was then heated to 750 K for periods of 12-24 hrs
and repeatedly flashed at higher temperature, up to 1500 k, for
a few seconds. The specimen undergoes further cleaning through

removal of several layers prior to initiation of successive adsorption observations. Finally the clean surface of the desired plane is photographically recorded and the imaging field is turned off. The Hf atoms are deposited by heating the evaporator while holding the specimen at 20 K. The imaging field is then reapplied to the tip and a sequence of photographic recordings of adatom position are taken as the field is increased through a careful combination of deposition and field desorption the presence of a single adatom on the plane is achieved.

For surface diffusion a plane of suitable size is obtained through field evaporation so that the edge effects are minimized and the brightness of the adatom at the center of the plane is adequate for clear observation of the adatom position. A single Hf atom on the plane as described above is obtained. The imaging field is then turned off and the specimen is then heated to a preset temperature for a fixed interval. After each heating interval the specimen is allowed to cool to 25 K to freeze out further thermal motion before the field is reapplied to register progress of diffusion. The same procedure is repeated more than 30 times at each temperature. Data at several temperatures is acquired to allow a determination of the mean square displacement as a function of temperature. The color superposition technique[4] is employed to measure displacements of the adatom from successive field ion micrographs. From the experimentally observed mean square displacement at various temperatures, the diffusion para-meters are obtained using the following equations.

$$D = D_o \exp(-V_D/RT) \quad , \quad D = \langle R^2\rangle/2d\tau$$

and
$$D_o = \frac{\nu \ell^2}{2d} \exp\left(\frac{\Delta S}{R}\right)$$

where $\langle R^2\rangle$ is the mean square displacement, V_D is the barrier to diffusion, k is the Boltzman constant, d the dimensionality of the walk, τ the heating time interval, ν the attempt frequency, ℓ the unit jump length and ΔS is the entropy of diffusion.

Following the methods of Ehlrich and Hudda[1] the edge correction to the mean square displacement due to finite size of the plane is approximated by

$$\langle r^2\rangle \approx N\ell^2 \left\{1-\frac{4}{3a}\left(\frac{2N\ell^2}{\pi}\right)^{1/2}\right\}$$

where a is the width of the plane and N is the observed number of jumps per heating interval.

Results and Discussion

Hafnium atoms deposited on W(111) were observed to desorb at 70-74% of the field corresponding to helium BIV. The quality of the image was found to be best when using a gas mixture consisting of He and 10% Ne rather than pure He or Ne. For direct determination of site geometry the use of sharp specimens of radius about 150Å and corresponding to a BIV about 6 kV or less is essential. A typical field ion micrograph showing a single Hf atom adsorbed on a 14 atom wide plane of W(111) is shown in Fig. 1 along with a hard sphere model of the plane. It can readily be seen that the Hf atom is sitting between three substrate atoms and occupied a normal lattice position. On larger planes the resolution is poor but the triangular symmetry at the adsorption site with verticies

pointing in the directions of the surrounding (211) planes is readily discernable. From a series of 115 well resolved individual adsorption events, it was concluded that in each case the Hf adsorption site geometry corresponded to normal, equilibrium, lattice site positions. The desorption field for Hf on W(111) of about 74% of the helium BIV is considerably smaller than 85% and 100-110% of the BIV in case of Ni and W adsorption on W(111) respectively[2,5]. The lower desorption field and larger atomic radii of Hf probably provides the resolution limit in the present images, which is somewhat worse than obtained in prior studies of Ni and W adsorption of W(111). The desorption field for Ni on W(111) is lower compared to W on W(111) and the single Ni atom diffusion onset occurred at lower temperature, about 385 K[2], compared to W on W(111) where the diffusion does not start before the specimen becomes contaminated. The lower desorption field in case of hafnium however is not associated with a decrease in the diffusion barrier. In fact, up to 600 k, the maximum temperature at which the tip remained free from contamination, no movement of the Hf atoms on the plane was observed. The binding of Hf on W(111) surface appears weaker as in the case of Ni compared to that of W but the diffusion barrier is comparable to W on W(111)[5].

The higher diffusion barrier in case of Hf on W(111) provides an opportunity to see if there is any affect of hydrogen on diffusion. Surprisingly in the presence of <1L H_2, the diffusion initiated at less than 500 k. The presence of hydrogen also reduces the desorption field from 74% of helium BIV to about 50%. The decrease in onset temperature of diffusion has also been

observed for single Ni atom self diffusion on Ni (110) and (311)[6].

Hf on W(211) and (321).

Both W(211) and (321) planes are channeled planes consisting of rows of closed packed atoms on hard sphere a model of these planes is shown in Fig. 2. Adatoms adsorbed on (211)W exhibit the same symmetry as the underlying substrate atoms.

Single Hf atom were deposited and more than 200 diffusion intervals were performed on both the (211) and (321) planes at the temperature indicated in Fig. 3. On W(211) plane the onset of single Hf atom diffusion was at 243 k with diffusion occurring along the channels. A diffusion sequence showing single Hf atom movement on (211) is shown in Fig. 4. The results are shown on the Arrhenius plot in Fig. 3. The measured activation energy V_D for single Hf atoms on W(211) is .54±.06 eV with a pre-exponential, $D_0 = 1.85 \times 10^{-6}$ cm^{2}s^{-1}. These studies show only channeled diffusion on W(211). A typical diffusion sequence of Hf on W(321) is shown in Fig. 4. The measured activation energy V_D for single Hf atom on W(321) is .57±.06 eV with a pre-exponential $D_0 = 4.0 \times 10^{-6}$ cm^{2}s^{-1}. Single Hf atom on both (211) and (321) planes exhibit normal diffusion characteristics. The pre-exponential $D_0 \sim 10^{-6}$ on both the planes is somewhat lower than the usual 10^{-1} to 10^{-5} cm^2/sec range. The activation energy for single Hf atom surface diffusion on both the (211) and (321) planes are lower than those reported[5,7] for W on these planes and in fact follow a decreasing trend from Re to Hf. This behavior is consistent with the trend observed for binding energy of 5d transition metal series on W planes[9]. The

lower activation energy in case of Hf thus appears to correlate with a decrease in available bonding electrons.

In conclusion we have observed that single Hf atom on W(111) plane adsorbs at normal, equilibrium lattice sites. The presence of hydrogen lowers the onset temperature for diffusion on this plane. Single Hf atom adsorbed on W(211) and (321) planes exhibit normal diffusion characteristics. The lower activation energy for Hf on W(211) and (321) planes follow a decreasing trend from Re to Hf and consistent with the trend reported for binding energies of 5d transition metals on W surfaces.

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Figure Captions

Fig. 1   (a) Micrograph and (b) hard sphere model of a W(111) specimen with a single Hf adatom adsorbed in the normal lattice site.

Fig. 2   Hard sphere model of the b.c.c. surface structure (a) 211 (b) 321.

Fig. 3   Diffusion sequence for single Hf adatom on W(211). The diffusion intervals were 20 sec long with the specimen heated to 295 k.

Fig. 4   Arrhenius plot of single atom hafnium diffusion on W(211) and (321) surfaces.

Fig. 5   Diffusion sequence for single Hf adatom on W(321). The diffusion intervals were 90 sec long with the specimen heated to 245 k.